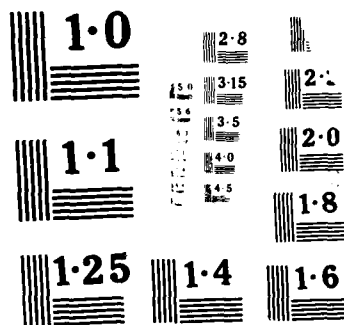


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**DEVELOPMENT OF A HIGH-TEMPERATURE RESISTANT (700°F),
CORROSION-PREVENTIVE ORGANIC COATING**

AD-A191 407

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Air Vehicle and Crew Systems Technology Department
NAVAL AIR DEVELOPMENT CENTER
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ABSTRACT (Cont.)

An organic coating has been developed that air dries at room temperature, is thermally stable up to 700°F and provides corrosion protection far superior to similar materials used for this type of application. The developed coating is based on a unique resin blend which can withstand exposure to 700°F. The blend of pigments used in this coating provides corrosion protection by both barrier and chemical means. This coating was initially designed to be used on steel aircraft engine components that are periodically subjected to temperatures in the 500°F to 700°F range. Additional testing, however, has shown that this coating can also be used on aluminum substrates.

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INTRODUCTION

Various areas of naval aircraft are routinely subjected to high temperatures. The epoxy primer (MIL-P-23377) and polyurethane topcoat (MIL-C-83286) paint system currently applied to Navy aircraft was designed to withstand exposures to temperatures up to 250°F for long durations (10 to 20 hours) and 350°F for short durations (1 to 2 hours). However, a survey of Naval Aviation Depots and airframe manufacturers indicated a need for a coating that would resist temperatures of 500 to 700°F and also protect the underlying metallic substrate from corrosion. One specific use for this material is on low carbon alloy steel heat shields around aircraft turbine engines. Currently, two materials are being used for this application: a silicone based aluminum paint covered under Federal specification TT-P-28, and Sermatel W, ceramic based material. Both of these materials have costly deficiencies. TT-P-28 can be spray applied, air-dries, and is thermally resistant. However, it provides poor corrosion protection and marginal film properties, therefore requiring frequent repair. Sermatel W is thermally resistant and provides good corrosion protection; but it requires a high-temperature cure and is very costly and difficult to apply. In addition, Sermatel W is extremely difficult to repair. Therefore, there is a need to find or develop a material for these types of application.

The coatings industry was surveyed for existing coatings that would provide the necessary temperature and performance requirements for this type of coating. Candidate materials were obtained from paint suppliers and evaluated. These materials proved to be inadequate for this application. Therefore, a new material had to be developed. To accomplish this task, samples of resin systems exhibiting good thermal properties were obtained. These materials were identified during the industry survey and consisted of silicones, silicone-copolymers, and polyimide resins. Along with these resins, thermally-stable pigments were also obtained. These materials were then used to formulate a coating with the desired properties. This report first describes the in-house development of this coating and then compares its performance with the current materials.

TEST METHODS

The substrates used to evaluate the properties of these coatings were: bare 1020 carbon steel (unpretreated), bare 2024-T3 aluminum alloy, and 2024-T0 aluminum alloy. The 2024-T3 aluminum panels were pretreated with a chromate conversion coating in accordance with specification MIL-C-5541 using materials conforming to specification MIL-C-81706. The 2024-T0 aluminum panels were anodized in accordance with specification MIL-A-8625, Type II. The coatings were applied by conventional air-spray to a dry-film thickness of 1.4 ± 0.4 mils. With the exception of the thermal cycling test, the physical properties of each coating were determined in accordance with test methods from the American Society for Testing and Materials (ASTM) and Federal Test Method Standard (FTMS) No. 141B.

1) Thermal Cycling Test

Since this type of coating would have to withstand repeated cyclic exposures to 700°F in service, a laboratory test method was developed that

simulated service conditions. Coated test panels were cyclically exposed for 8 hours at 700°F and then 16 hours at room temperature (70°F). This cycle was repeated five times for a total test time of 120 hours (40 hours at 700°F total). The test panels were evaluated at the end of each test cycle for blistering, uplifting, or other forms of coating failure. The coatings were rated using a system that correlated to the length of time before failure in the thermal stability test. The rating scale ranged from 1 to 10; where 1 corresponded to failure during the first cycle (<1 day), 2 failed between the first and second cycles, and so on up to 10 which passed all 5 days of exposure. Another less severe, thermal cycling test was developed for comparison of the final coating properties with those of the current materials. The cycle in this test consisted of 5 hours at 400°F, 3 hours at 500°F, and 16 hours at room temperature and was also repeated five times.

2) Drying Time (ASTM D 1640)

Dry-hard time of the coating was measured and is defined as the period of time from application until the film cannot be permanently marred using firm thumb pressure. At this point, a painted component can be handled without causing permanent damage to the coating.

3) Adhesion (FTMS 6301, ASTM D 2197A)

Adhesion to steel and aluminum substrates was measured by the tape test and the scrape-adhesion test. For the tape test, an "X" was scratched between two parallel, scribed lines approximately one-inch apart on the test panel. Then, a strip of 3M-250 masking tape was applied over the scribe marks with firm thumb pressure and removed with one quick motion. Removal of the coating from the substrate is grounds for failure. This test was performed on both dry specimens and on specimens that were immersed in water at 70°F for 24 hours. For the scrape-adhesion test, a weighted stylus was scraped across the test panel from bare substrate onto a coated section. The minimum weight to cut through the coating to the substrate is the scrape-adhesion value.

4) Chemical Resistance

Chemical resistance was evaluated by exposing coated panels to various fluids for a period of one day. Resistance to hydraulic fluids and lubricating oils was determined by immersing coated panels in MIL-H-83282 hydraulic fluid at 150°F and MIL-L-23699 lubricating oil at 250°F. The coatings were then observed for softening or other signs of film degradation. Resistance to hydrocarbons was ascertained by rubbing cloth rags soaked in methyl ethyl ketone (MEK) and MIL-T-5624, Grade JP-4 aviation fuel back and forth across the surface of the panel ten times. Complete removal of the coating from the panel represented failure in these tests. Water resistance of the coatings was determined by immersing coated test specimens in distilled water at room temperature for 24 hours. The panels were removed and immediately examined for signs of blistering or softening of the coating.

5) Impact Flexibility (FTMS 6226)

Impact resistance of the coatings was measured by means of a GE impact tester. This test was performed on the anodized, 2024-T0 aluminum alloy

panels. The results are given as the maximum percentage elongation that the coating can withstand without cracking.

6) Corrosion Resistance (ASTM B 117)

Coated panels were thermally aged, scribed with an "X" down to bare substrate on the bottom one-third of the panel and mounted in a 15 degree rack. These panels were then exposed to a 5% NaCl salt spray environment. Periodically these panels were removed, and the surfaces were evaluated for evidence of corrosion products to determine the corrosion resistance of the coating.

7) Pencil-Hardness Test (ASTM D 3363)

The pencil-hardness of a coating gives an indication of the film's integrity. The hardness value is obtained by firmly holding the pencil against the surface at a 45-degree angle and pushing across the surface in an even stroke. The hardness rating of the pencil that does not permanently scratch the surface is the pencil-hardness of the coating.

DISCUSSION OF COATING DEVELOPMENT

Development of the final coating was performed in three stages: resin system development, pigment selection and final coating development.

Resin System Development

The first stage in this process was the development of a thermally-stable resin system with the appropriate binder properties (i.e. adhesion, drying characteristics, film integrity, etc.). The requirements for this binder system were: 1) The coating must dry tack-free at room temperature (70°F) in less than eight hours with sufficient integrity to be handled without inflicting permanent damage; 2) The coating must be adherent to both steel and aluminum substrates; and 3) The coating must maintain its integrity up to 700°F for prolonged periods (eight hours) and multiple thermal cycles. Candidate resins investigated for this coating consisted of polysiloxanes, silicone copolymers, and polyimides. To screen these materials, the resins were applied to several steel panels as clear coatings and allowed to air-dry for a minimum of 48 hours. Then, these panels were subjected to the 70 to 700°F thermal cycling test which provided information on how well the unpigmented material performed when subjected to high temperatures and thermal fatigue. In addition, the film properties (i.e. adhesion, drying time, and pencil hardness) of the candidate binders were analyzed both before and after thermal cycling.

After initial screening, the polyimide resins were deleted from further investigation for several reasons. First, they would not form clear homogeneous films when applied to the steel test panels. Even the addition of surface active agents did not solve this non-uniformity problem. Second, the resultant films would not dry-hard. After several days, they still remained tacky. Finally, after exposure to thermal cycling, the coatings were brittle and filled with air-voids.

All of the silicone and silicone copolymer resins formed clear homogeneous coatings when spray applied to the test panels. However, they all lacked at least one or more of the required binder properties. Table I shows the specific properties of the materials tested. The silicone copolymers had good room-temperature coatings properties. However, they lacked thermal stability. The silicones could be grouped into two categories. Although some resins were thermally stable, they remained tacky at room temperature with no film integrity and had only marginal film properties after thermal cycling. The rest of the resins had good thermal stability and were dry-hard at room temperature, but had poor coatings properties both before and after thermal cycling. Since no individual resin could provide the required binder properties, polymer-blend technology was incorporated to provide an appropriate binder system. The initial test results, along with information provided in the product literature, were used to choose the optimum materials for the polymer-blend formulations. Three resins were chosen for further binder development. The McCloskey 385-50E silicone-alkyd resin was selected because it had the best room-temperature film properties; although it lacked thermal stability. The G.E. SR 125 silicone resin was chosen because it performed the best in the thermal cycling test. Even though it remained tacky at room temperature, the SR 125 resin cured to a flexible film during thermal cycling. The G.E. SR 141 silicone dried at room temperature, forming a very brittle film. This material had good thermal resistance, but only survived one day of thermal cycling. This failure was attributed to the brittle nature of the coating. Combining this resin with the SR 125 and the 385-50E resins would enhance both the high-temperature resistant and the as-applied properties of the coating.

A simplex-design statistical screening method was used to obtain the polymer-blend formulations (reference (1)). Figure 1 is a tri-coordinate graph showing the first series of resin blends. Once prepared, these resin combinations were thinned to spray viscosity with toluene and applied to steel panels as clear coatings to a dry-film thickness of 1.0 to 1.5 mils. After 48 hours at room temperature, the coated panels were subjected to the 70 to 700°F thermal cycling test. The coatings were rated using a scale from 1 (worst) to 10 (best). This rating system correlated to the length of time before failure in the thermal stability test. Table II contains the first series of resin blend formulations and their thermal cycling ratings. From these results, a composition region of thermal stability was identified. This region is shown in Figure 2. The next series of formulations were prepared from this region and tested to determine their thermal stability (see Table III). The results from the second series of formulations narrowed down the region of thermal stability to a specific area. The last series of resin formulations was taken from this area. This information appears in Table IV and Figure 3. All of the resin systems tested in this area passed five days in thermal cycling. The drying characteristics and film integrity of all the resin blends that rated a ten in the thermal cycling test were then evaluated; and five potential systems were identified. They were formulations R-08, R-15, R-22, R-24, and R-25. These systems and were used in the final coating development.

Pigment Selection

Pigment selection consisted of first determining a pigment system that was thermally stable up to 700°F, in addition to providing the required corrosion protection. The initial screening of candidate pigments was

TABLE I CANDIDATE RESIN PROPERTIES

RESIN ID	RESIN TYPE	DRYING TIME		THERMAL STABILITY		PENCIL HARDNESS (4B..B,H..4H) (1DAY) (1WEEK)	ADHESION DRY TAPE (>1WEEK)
		TACK-FREE (HOURS)	DRY-HARD (HOURS)	(5 DAY-70 TO 700 F CYCLE) PANEL #1	PANEL #2		
GEN. ELEC. 112	PURE SILICONE	>168 HRS	----	4 DAYS	3 DAYS	--	----
GEN. ELEC. 125	PURE SILICONE	8.0 HRS	>168 HRS	4-5 DAYS	5 DAYS	<4B	PASS
GEN. ELEC. 141	PURE SILICONE	1.0 HRS	2.0 HRS	1-2 DAYS	1-2 DAYS	3B	PASS
GEN. ELEC. 240	PURE SILICONE	2.0 HRS	4.0 HRS	<1 DAY	<1 DAY	<4B	PASS
GEN. ELEC. 882	PURE SILICONE	>168 HRS	----	<1 DAY	<1 DAY	--	----
CARGILL 6260	PURE SILICONE	>168 HRS	----	<1 DAY	<1 DAY	--	----
CARGILL 6247	SIL.-EPOXY	0.5 HRS	1.0 HRS	<1 DAY	<1 DAY	<4B	PASS
MCCLOSKEY 385-50E	SIL.-ALKYD	0.5 HRS	1.5 HRS	<1 DAY	<1 DAY	<4B	PASS

FIGURE 1. TRICOORDINATE GRAPH WITH FIRST RESIN BLEND SERIES.

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TABLE II FIRST RESIN BLEND SERIES

RESIN FORMULATION NO.	SR141 RESIN PARTS	SR125 RESIN PARTS	385-50E RESIN PARTS	THERMAL STABILITY TEST 5 DAY-70 TO 700F CYCLE	
				PANEL #1	PANEL #2
R - 01	---	100.0	---	8	10
R - 02	100.0	---	---	2	2
R - 03	50.0	50.0	---	2	4
R - 04	---	50.0	50.0	10	10
R - 05	50.0	---	50.0	1	1
R - 06	33.3	33.3	33.3	2	3
R - 07	66.7	16.6	16.6	6	4
R - 08	16.6	66.7	16.6	10	10
R - 09	16.6	16.6	66.7	1	1
R - 10	---	---	100.0	1	1

THERMAL STABILITY RATING SYSTEM:

1=<1 DAY, 2=1-2 DAYS...10=>5 DAYS

TABLE III SECOND RESIN BLEND SERIES

RESIN FORMULATION NO.	SR141 RESIN PARTS	SR125 RESIN PARTS	385-50E RESIN PARTS	THERMAL STABILITY TEST 5 DAY-70 TO 700F CYCLE	
				PANEL #1	PANEL #2
R - 11	40.0	40.0	20.0	5	2
R - 12	10.0	35.0	55.0	1	1
R - 13	---	75.0	25.0	10	10
R - 14	25.0	75.0	---	2	2
R - 15	10.0	80.0	10.0	10	10
R - 16	22.5	55.0	22.5	9	10
R - 17	5.0	60.0	35.0	10	10
R - 18	15.0	45.0	40.0	3*	10

THERMAL STABILITY RATING SYSTEM:

1=<1 DAY, 2=1-2 DAYS...10=>5 DAYS

* 5-10% FAILURE AT
PREEXISTING FLAW

TABLE IV THIRD RESIN BLEND SERIES

RESIN FORMULATION NO.	SR141 RESIN PARTS	SR125 RESIN PARTS	385-50E RESIN PARTS	THERMAL STABILITY TEST 5 DAY-70 TO 700F CYCLE	
				PANEL #1	PANEL #2
R - 21	20.0	70.0	10.0	10	10
R - 22	10.0	70.0	20.0	10	10
R - 23	10.0	65.0	25.0	10	10
R - 24	---	85.0	15.0	10	10
R - 25	10.0	75.0	15.0	10	10

THERMAL STABILITY RATING SYSTEM:

1=<1 DAY, 2=1-2 DAYS...10=>5 DAYS

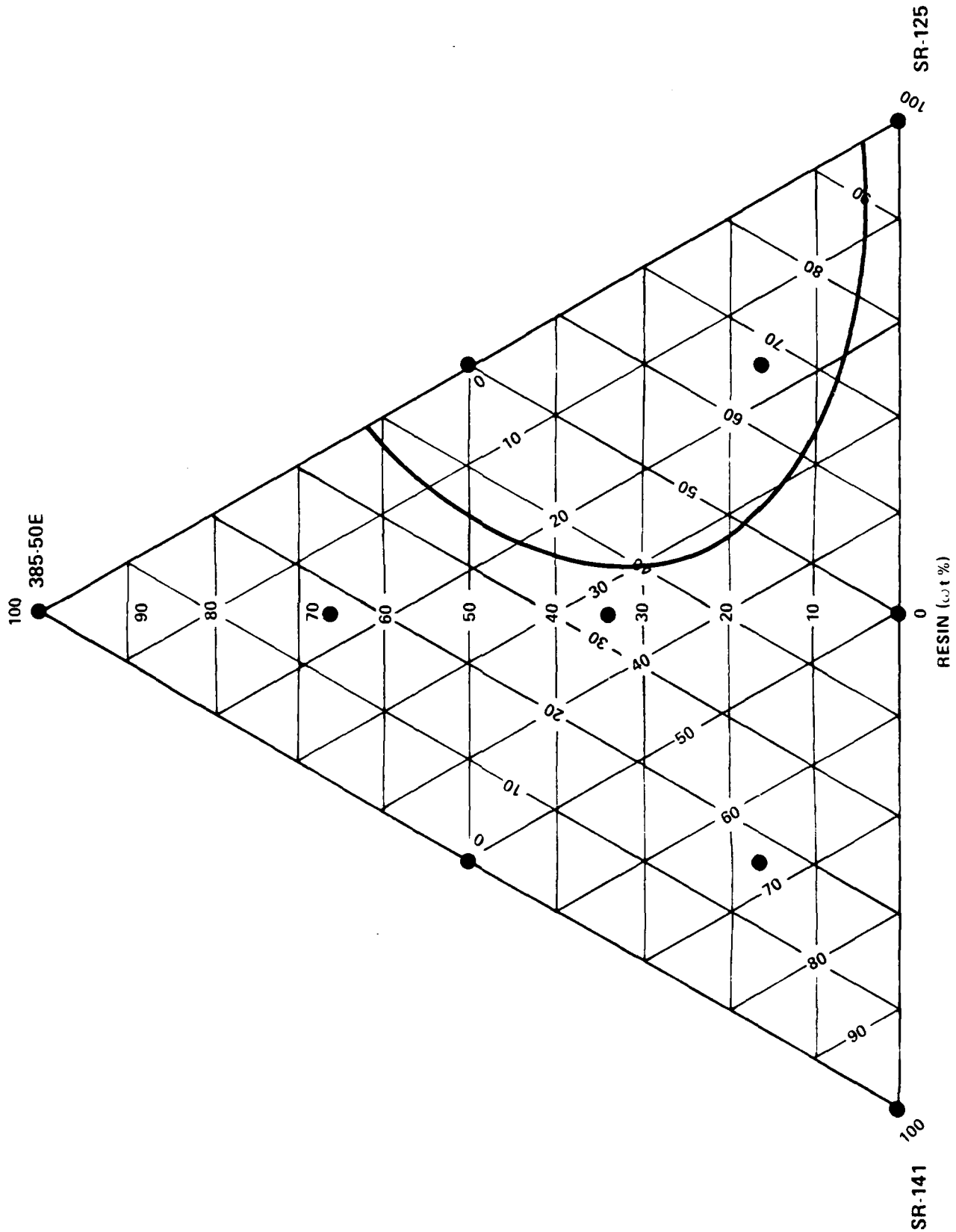


FIGURE 2. GRAPH SHOWING REGION OF THERMAL STABILITY.

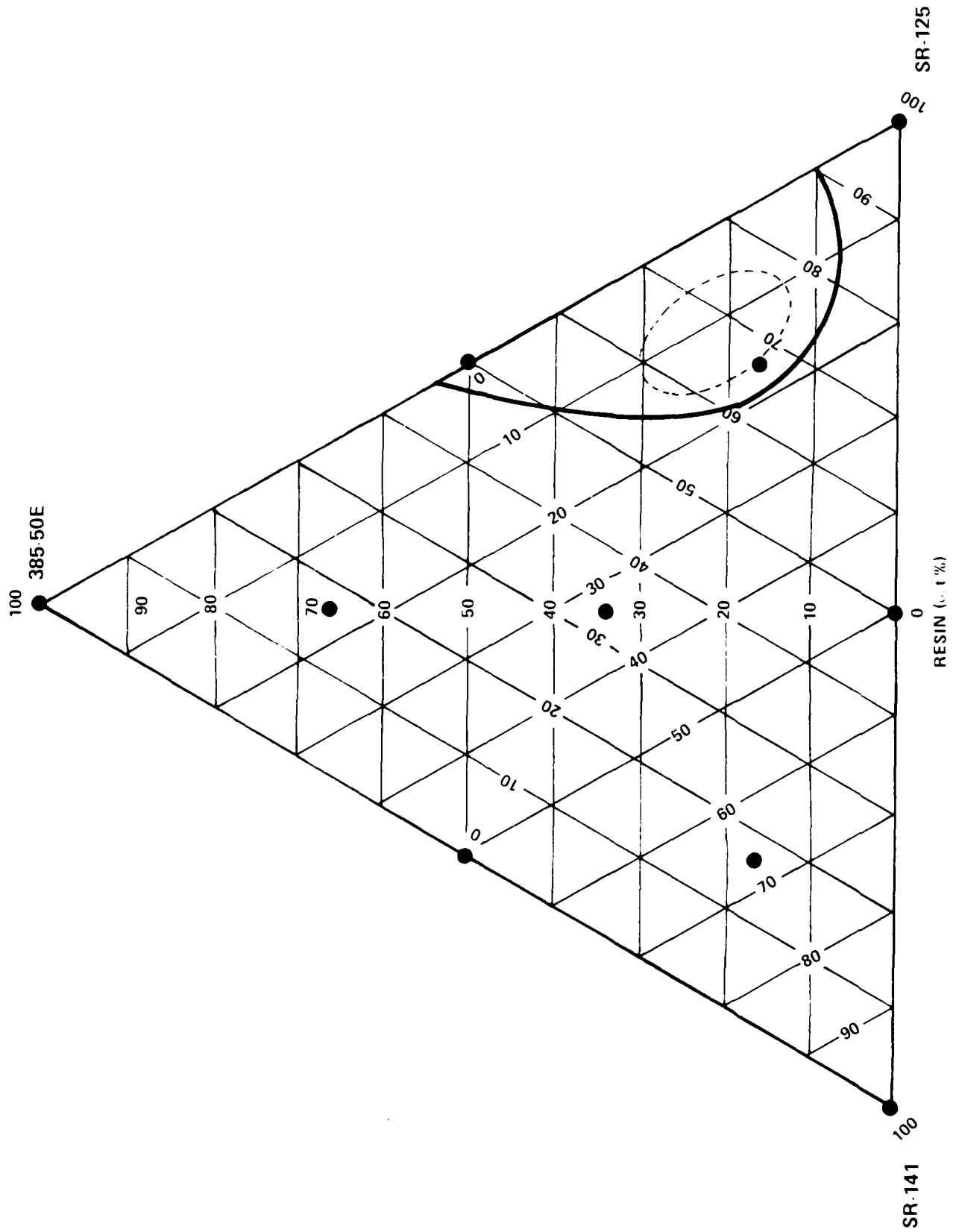


FIGURE 3. SPECIFIC AREA OF THERMAL STABILITY.

accomplished solely by thermal analysis. Both Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) were performed on the potential materials to determine their thermal stability. Table V lists the thermally stable pigments identified from this testing. These pigments were incorporated into coating formulations obtained from a computer program that calculates theoretical optimum coating formulations based on oil absorptions and pigment packing factors. This formulating procedure is described in detail in reference (2). Using these formulations, the coatings were prepared with the resin systems developed above. The pigments were milled into the resin systems using ball/shot milling techniques, with the exception of the leafing aluminum pigment. This material attains the leafing action by its platelet shape which would be destroyed by ball milling. Therefore mechanical stirring was used to disperse this pigment. Once prepared, all of these coatings were applied to steel panels and analyzed for thermal, mechanical, chemical and corrosion inhibiting properties.

Three pigments were eliminated at this point. Titanium dioxide coatings provided only minimal protection against the corrosion process as a barrier coating; and therefore, were dropped from further consideration in this study. Red iron oxide coatings tended to crack and uplift some time after the completion of the thermal cycling test, which resulted in poor performance in the corrosion resistance tests. Further experimentation with red iron oxide coatings with lower pigment volume concentrations still resulted in poor performance in these tests. Consequently, this pigment was also dropped from the investigation. Finally, the leafing aluminum pigment performed far better than the particulate aluminum pigment in these initial coatings. Therefore, the aluminum powder was disregarded in favor of the leafing pigment.

Finally, the two zinc-dust pigments were analyzed and compared in all five resin systems. In most of these tests, there was little difference between the two materials. However, the Zn101 performed better than the Zn303 in the corrosion resistance tests and therefore, was used for the final coating development.

Final Coating Development

To facilitate in the development of the final coating system only the #25 resin system was used with the two pigment types (zinc dust and leafing aluminum). This resin blend was selected because it was centrally located in the area outlined by the five approved resin systems.

The optimum pigment volume concentrations (PVC's) for the zinc-dust and the leafing aluminum pigments had to be determined. Formulations using the theoretical prediction program stated earlier were used as upper boundary limits for a multi-level factorial design using pigment type and solvent type versus PVC. Table VI shows this factorial design. The formulations for these coatings are listed in Table VII and the test results of these materials appear in Tables VIII & IX.

Analysis of the data in Tables VIII and IX for the zinc-dust coatings showed that the optimum properties for this pigment were obtained using a PVC of 40%. Above this PVC, the physical properties began to deteriorate; and, below this PVC the corrosion resistance properties were significantly decreased. Also, these test results indicated that there was virtually no

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TABLE V THERMALLY STABLE PIGMENTS

PIGMENT TYPE	PIGMENT MANUFACTURER	PIGMENT IDENTIFICATION
RED IRON OXIDE	PFIZER CO.	#R-2199
ALUMINUM POWDER	ALCAN POWDERS AND CHEMICALS	AL. POWDER #9900
LEAFING ALUMINUM	ALCAN POWDERS AND CHEMICALS	AL. FLAKE #515
TITANIUM DIOXIDE	E. I. DUPONT INC.	#R-960
ZINC DUST	LDL TECHNOLOGY INC.	ZN-101 AND ZN-303

TABLE VI STATISTICAL DESIGN FOR OPTIMUM PVC DETERMINATION

PIGMENT VOLUME CONCENTRATION	POLAR SOLVENTS PIGMENTS		NON-POLAR SOLVENTS PIGMENTS	
	ZN-101	ALCAN #515	ZN-101	ALCAN #515
60	X	X	X	X
50	X	X	X	X
40	X	X	X	X
30	X	X	X	X
20	X	X	X	X
10	X	X	X	X

TABLE VII PVC COATING SERIES FORMULATIONS

NADC FORMULATION NO. 8372-	SOLVENT POLARITY	RESINS				PIGMENTS			SOLVENTS			PIGMENT VOLUME CONC.
		SR141	GE	PARTS BY WEIGHT 385- 50E	ZN101	ALCAN 515	TOLUENE	MEK	CELLOSOLVE ACETATE			
46-Z1-25-A6	POLAR	1.9	14.0	2.8	81.3	---	25	25	30	60		
50-Z1-25-A5	POLAR	2.6	19.2	3.8	74.4	---	30	20	30	50		
50-Z1-25-A4	POLAR	3.4	25.5	5.1	66.0	---	30	20	30	40		
57-Z1-25-A3	POLAR	4.5	33.3	6.7	55.5	---	15	5	10	30		
61-Z1-25-A2	POLAR	5.8	43.2	8.7	42.3	---	15	5	10	20		
61-Z1-25-A1	POLAR	7.6	56.6	11.3	24.5	---	15	5	10	10		
46-A5-25-A5	POLAR	3.6	27.3	5.5	---	63.6	25	25	30	50		
50-A5-25-A4	POLAR	4.6	34.7	6.9	---	53.8	30	20	30	40		
71-A5-25-A3	POLAR	7.2	53.9	10.8	---	28.1	15	5	10	30		
46-Z1-25-A6	NON-POLAR	1.9	14.0	2.8	81.3	---	50	---	30	60		
50-Z1-25-A5	NON-POLAR	2.6	19.2	3.8	74.4	---	50	---	30	50		
50-Z1-25-A4	NON-POLAR	3.4	25.5	5.1	66.0	---	50	---	30	40		
57-Z1-25-A3	NON-POLAR	4.5	33.3	6.7	55.5	---	20	---	10	30		
61-Z1-25-A2	NON-POLAR	5.8	43.2	8.7	42.3	---	20	---	10	20		
61-Z1-25-A1	NON-POLAR	7.6	56.6	11.3	24.5	---	20	---	10	10		
46-A5-25-A5	NON-POLAR	3.6	27.3	5.5	---	63.6	50	---	30	50		
50-A5-25-A4	NON-POLAR	4.6	34.7	6.9	---	53.8	50	---	30	40		
71-A5-25-A3	NON-POLAR	7.2	53.9	10.8	---	28.1	20	---	10	30		

TABLE VIII PVC COATING SERIES PROPERTIES

NADC FORMULATION NO. 8372-	DRYING TIME DRY-HARD (Hrs)	ADHESION TESTS		THERMAL CYCLING (5 Days @ 700F)	FLEXIBILITY GE IMPACT %elongation	FLUID RESISTANCE			
		DRY TAPE	WET TAPE			JP-5 FUEL (77 F)	MIL-H-83282 (150 F)	MIL-L-23699 (250 F)	DI-WATER (77 F)
46-Z1-25-A6	P - 1.0	P	P	P	5	P	P	P	P
50-Z1-25-A5	P - 1.0	P	P	P	10	P	P	P	P
50-Z1-25-A4	P - 1.0	P	P	P	40	P	P	P	P
57-Z1-25-A3	P - 1.0	P	P	P	60	P	P	P	P
61-Z1-25-A2	P - 1.0	P	P	P	60	P	P	P	P
61-Z1-25-A1	P - 1.0	P	P	P	60	P	P	P	P
46-A5-25-A5	P - 1.0	P	P	P	10	P	P	P	P
50-A5-25-A4	P - 1.0	P	P	P	20	P	P	P	P
71-A5-25-A3	P - 1.0	P	P	P	20	P	P	P	P
46-Z1-25-A6	NP - 2.0	P	P	P	5	P	P	P	P
50-Z1-25-A5	NP - 2.0	P	P	P	10	P	P	P	P
50-Z1-25-A4	NP - 2.0	P	P	P	40	P	P	P	P
57-Z1-25-A3	NP - 2.0	P	P	P	60	P	P	P	P
61-Z1-25-A2	NP - 2.0	P	P	P	60	P	P	P	P
61-Z1-25-A1	NP - 2.0	P	P	P	60	P	P	P	P
46-A5-25-A5	NP - 2.0	P	P	P	10	P	P	P	P
50-A5-25-A4	NP - 2.0	P	P	P	20	P	P	P	P
71-A5-25-A3	NP - 2.0	P	P	P	40	P	P	P	P

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TABLE IX PVC COATING SERIES CORROSION RESISTANCE PROPERTIES
ASTM #B 117 (5% NaCl SALT SPRAY TEST)

FORMULATION NO. 8372-	STEEL (500 HOURS) SCRIBE AREA	STEEL (500 HOURS) GENERAL COATING SURFACE
46-Z1-25-A6 (P) (Polar solv.)	1 , 2	White residue over panel with a few spots of corrosion on panel
50-Z1-25-A5 (P) (Polar solv.)	1 , 2	White residue over panel with about 5 spots of corrosion on panel
50-Z1-25-A4 (P) (Polar solv.)	2 , 3	White residue over panel with about 20 small spots of corrosion on panel
57-Z1-25-A3 (P) (Polar solv.)	4 - WR	Approximately 50 spots of corrosion with rundown over 75% of panel
61-Z1-25-A2 (P) (Polar solv.)	5 - WHR	Many spots of corrosion on surface with heavy rundown over most of panel
61-Z1-25-A1 (P) (Polar solv.)	5 - WHR	Many spots of corrosion on surface with heavy rundown over most of panel
46-A5-25-A5 (P) (Polar solv.)	4 - WR	Approx. 50 small spots of corrosion over panel surface with rundown
50-A5-25-A4 (P) (Polar solv.)	5 - WR	Approx. 100 small spots of corrosion over panel surface with rundown
71-A5-25-A3 (P) (Polar solv.)	5 - WHR	Many spots of corrosion on surface with heavy rundown over most of panel
46-Z1-25-A6(NP)	1 , 2	Same as polar solvent results above
50-Z1-25-A5(NP)	1 , 2	Same as polar solvent results above
50-Z1-25-A4(NP)	2 , 3	Same as polar solvent results above
57-Z1-25-A3(NP)	4 - WR	Same as polar solvent results above
61-Z1-25-A2(NP)	5 - WHR	Same as polar solvent results above
61-Z1-25-A1(NP)	5 - WHR	Same as polar solvent results above
46-A5-25-A5(NP)	4 - WR	Approx. 100 spots of corr. w/ rundown
50-A5-25-A4(NP)	5 - WHR	Approx. 200 spots of corr. w/ rundown
71-A5-25-A3(NP)	5 - WHR	Same as polar solvent results above

SCRIBE RATINGS:

- 1 - No corrosion in scribe 2 - Slight corrosion in scribe
 3 - Some corrosion in scribe 4 - Moderate corrosion in scribe
 5 - Heavy corrosion in scribe
 WR - With rundown from scribe WHR - With heavy rundown from scribe

difference between the zinc-dust coatings with the polar solvents and the non-polar system.

Determination of the optimum PVC for the leafing aluminum pigment involved experimentation with the solvent system as well as the PVC. The leafing efficiency of the pigment is a function of the wettability of the pigment by the resin in addition to the type and amount of solvent. The leafing nature of the material occurs by the alignment of the pigment in the resin to form a pseudolayer of aluminum (reference (3)). The alignment of the aluminum platelets is enhanced by the incompatibility of the resin with the pigment and the driving force is provided by the solvent evaporation. The long term stability of the pigment requires a non-polar solvent system like the aromatic hydrocarbons; however, the leafing action performs better in polar solvents like ketones and alcohols. Therefore, the coating formulation must consist of only non-polar solvents, while at the time of application, the material is thinned with polar solvents. Using this concept, the optimum PVC for the aluminum was determined to be 40%. These formulations and test results also appear in Tables VII, VIII, & IX.

The final experimental zinc and aluminum coatings were compared to samples of TT-P-28. This information is shown in Tables X & XI. During the thermal cycling test one of the TT-P-28 coatings failed and was dropped from further testing. The results for the rest of the physical tests indicated that the experimental coatings performed slightly better than the currently-used materials. The salt-spray corrosion test results also showed that the zinc coatings performed better than the TT-P-28 and the performance of the aluminum coatings was comparable to the standard coatings. When evaluating the test panels with the zinc dust, there was a white residue on the surface of the panels. This product is ZnO and is the result of the sacrificial chemical nature of the zinc pigment. The aluminum coatings had small areas where the coating failed, but provided good barrier protection over the rest of the panel surface.

The zinc-dust provided good chemical protection to the substrate and as stated previously, the aluminum pigment provided good barrier protection. Based on these two observations, it was decided to try both pigments in the same coating. The pigments were mixed in volume ratios of 3:1, 1:1 and 1:3; at PVC levels of 30% and 40%. The information for these coatings appears in Tables XII, XIII, & XIV. As stated before, a non-polar solvent system was used for the coating formulations; and, polar solvents were used to thin for application. General film properties for these formulations were similar, providing little distinction between these coatings and the previous materials. However, the corrosion resistance properties of the binary pigment system coatings were far better than those for the individually pigmented coatings. Further analysis of these results showed that the 1/1 pigment volume ratio with a total PVC of 40% was the optimum coating formulation.

The final coating was prepared by first milling the zinc-dust pigment in the resin system using only non-polar solvents. Then, the leafing aluminum pigment was mechanically stirred into the coating. Finally, when applying the coating using conventional air-spray, a polar solvent system was used to thin the material, while continually being mechanically stirred.

TABLE X COMPARISON OF SINGLE PIGMENT COATING PROPERTIES

NADC IDENTIFICATION NUMBER	DRY-HARD TIME (Hrs)	ADHESION DRY/WET TAPE	TESTS SCRAPE (Kgrms)	THERMAL		GE IMPACT FLEXIBILITY %elongation	FLUID RESISTANCE			
				CYCLING (5 Days @ 700F)	F		P	JP-5 FUEL (77 F)	MIL-H-83282 (150 F)	MIL-L-23699 (250 F)
TT-P-28-#1	2.5	P / F	1.0	F		1	P	P	P	F
TT-P-28-#2	1.25	P / P	<.5	P		1	P	P	P	P
FORMULATION NO. 8372- 50-Z1-25-A4	1.0	P / P	1.0	P		40	P	P	P	P
FORMULATION NO. 8372- 50-A5-25-A4/P	1.0	P / P	1.0	P		20	P	P	P	P

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TABLE XI SINGLE PIGMENT COATINGS CORROSION RESISTANCE PROPERTIES
ASTM METHOD #B 117 - 5% NaCl SALT SPRAY TEST
UNPRETREATED STEEL SUBSTRATE - 500 HOURS

COATING ID	SCRIBE AREA	GENERAL COATING SURFACE
TT-P-28-#1	5 - WHR	Many spots of corrosion on surface with heavy rundown, in addition to heavily corroded areas where the coating lifted
TT-P-28-#2	5 - WHR	Many spots of corrosion with heavy rundown over the surfaces of both test panels
FORMULATION NO. 8372- 50-Z1-25-A4	2 , 3	White residue over the surfaces of both panels with approximately 20 small spots of corrosion on panel
FORMULATION NO. 8372- 46-A5-25-A5(P)	5 - WR	Approximately 100 very small spots of corrosion on the surfaces of both panels with some slight rundown

SCRIBE RATINGS:

- | | |
|-------------------------------|--------------------------------------|
| 1 - No corrosion in scribe | 2 - Slight corrosion in scribe |
| 3 - Some corrosion in scribe | 4 - Moderate corrosion in scribe |
| 5 - Heavy corrosion in scribe | |
| WR - With rundown from scribe | WHR - With heavy rundown from scribe |

TABLE XII ZINC DUST/ALUMINUM COATING SERIES FORMULATIONS

NADC FORMULATION NO. 8372-	RESINS			PIGMENTS		SOLVENTS		PIGMENT VOLUME CONC.
	SR141	SR125	385-50E	ZN101	ALCAN 515	TOLUENE	MEK CELLOSOLVE ACETATE	
64-A/Z-25-3/1	4.2	31.8	6.4	20.6	37.0	10	3 6	40
64-A/Z-25-2/2	3.9	29.4	5.9	38.0	22.8	10	3 6	40
64-A/Z-25-1/3	3.6	27.3	5.5	53.0	10.6	10	3 6	40
71-Z/A-25-3/1	4.9	36.9	7.4	46.0	4.8	10	--- 8	30
71-Z/A-25-2/2	5.5	41.2	8.2	34.3	10.8	10	--- 8	30
71-Z/A-25-1/3	6.2	46.7	9.4	19.4	18.3	10	--- 8	30

TABLE XIII ZINC DUST/ALUMINUM COATING SERIES PROPERTIES

NADC FORMULATION NO. 8372-	DRY-HARD TIME (Hrs)	ADHESION TESTS		THERMAL CYCLING (5 Days @ 700F)	GE IMPACT FLEXIBILITY elongation (%)	FLUID RESISTANCE			
		DRY/WET TAPE	SCRAPE (Kgs)			JP-5 FUEL (77 F)	MIL-H-83282 (150 F)	MIL-L-23699 (250 F)	DI-WATER (77 F)
64-A/Z-25-3/1	1.0	P / P	1.0	P	60	P	P	P	P
64-A/Z-25-2/2	1.0	P / P	1.0	P	60	P	P	P	P
64-A/Z-25-1/3	1.0	P / P	1.0	P	40	P	P	P	P
71-Z/A-25-3/1	1.0	P / P	1.0	P	60	P	P	P	P
71-Z/A-25-2/2	1.0	P / P	1.0	P	60	P	P	P	P
71-Z/A-25-1/3	1.0	P / P	1.0	P	60	P	P	P	P

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TABLE XIV ZINC DUST/ALUMINUM COATINGS CORROSION RESISTANCE PROPERTIES
ASTM METHOD #B 117 - 5% NaCl SALT SPRAY TEST
UNPRETREATED STEEL SUBSTRATE - 500 HOURS

FORMULATION NO. 8372-	SCRIBE AREA	GENERAL COATING SURFACE
64-A/Z-25-3/1	3 - WR	A number of small spots of corrosion on the surfaces of both panels with some slight rundown
64-A/Z-25-2/2	1	No evidence of any corrosion on the coating surface of either test panel
64-A/Z-25-1/3	2	Some small spots of corrosion on the surfaces of both panels with a slight trace of some rundown
71-Z/A-25-3/1	2	Some small spots of corrosion on the surfaces of both panels with a slight trace of some rundown
71-Z/A-25-2/2	3 - WR	Only a few spots of corrosion on the coating surface of both test panels
71-Z/A-25-1/3	4 - WR	Many small spots of corrosion on the surfaces of both panels with some slight rundown

SCRIBE RATINGS:

- 1 - No corrosion in scribe
- 2 - Slight corrosion in scribe
- 3 - Some corrosion in scribe
- 4 - Moderate corrosion in scribe
- 5 - Heavy corrosion in scribe
- WR - With rundown from scribe
- WHR - With heavy rundown from scribe

DISCUSSION OF COMPARISON OF HTC'S PERFORMANCE

The final coating formulations were evaluated against several high temperature coatings, consisting of two samples of TT-P-28 and two high temperature coatings (HTC) submitted from industry. The properties of these materials were compared after exposure to four different thermal conditions. These thermal conditions were: the five day 70 to 700°F cycle; the five day 70 to 400 to 500°F cycle; one hour at 700°F; and seven days at room temperature (77°F). Standard coating tests were performed on coated steel test panels and this information appears in Tables XV to XXII.

Physical test results for the coatings exposed to the 70 to 700°F cycle are summarized in Table XV. Some of the specimens for the TT-P-28-#1 sample failed the thermal cycling test. This coating also failed the water resistance and wet tape adhesion tests. The other sample of TT-P-28, however, passed all of these tests. This indicated a possible quality control problem with this specification material. Further analysis of the test data showed that while all the coatings passed the thermal cycling, tape adhesion and fluid resistance tests, the developed coating dried faster, had a higher impact flexibility rating and better scrape adhesion than the TT-P-28 coatings and the industry HTC's. Salt spray tests were conducted on painted steel panels for 500 hours. See Table XVI for these results. The scribe areas of the experimental coatings showed little to no evidence of corrosion, while the rest of the specimens had heavy corrosion throughout the scribe area with heavy rundown of corrosion products from the scribe. The results for the general surface area also showed significant differences in performance between the coatings. The industry HTC's were as badly corroded as the uncoated blank panels. TT-P-28 panels had many spots of corrosion over the surface with excessive rundown from the spots, covering the majority of the panel. The developed coatings, however, had only a few spots of corrosion on some panels and none on the panels with the #25 resin system. Figure 4 shows the test specimens after salt spray exposure illustrating these differences.

Tables XVII and XVIII contain the data for the one hour at 700°F exposure condition. Again, the TT-P-28-#1 sample failed the thermal conditioning, and therefore, no further testing was performed on these specimens. The other TT-P-28 material failed both dry and wet tape adhesion tests and performed poorly in the scrape adhesion and impact flexibility tests. The HTC-1 passed the tape adhesion tests, but performed similar to the TT-P-28-#2 in the other tests. Under this condition, the experimental coatings were slightly less flexible than those obtained under the five day 700°F cycle. In addition, there was removal of very small areas at the films surface in some of the tape tests. Since this removal occurred only at the surface of the film, it was not significant enough to be considered a failure. The remaining properties of the coatings under this thermal exposure were the same as those conditioned under the five day 700°F cycle. Finally, the HTC-2 performed slightly better than the in-house coatings in the flexibility and tape tests and similarly in the rest of the physical tests. All of the coatings passed the fluid resistance tests. The corrosion resistance test results showed that the TT-P-28 and the HTC's performed as poorly as they did under the more severe thermal conditioning above. The NADC coatings, in these tests, displayed better performance than the other materials with only a slight decrease in the quality of protection provided as compared to the specimens exposed to the 70 to 700°F condition.

TABLE XV COMPARISON OF HIGH TEMPERATURE COATINGS (5 DAY - 700F Cycle)

COATING IDENTIFICATION	DRY-HARD TIME (Hrs)	THERMAL CYCLING (5 Days @ 700F)	ADHESION TESTS DRY/WET TAPE	SCRAPE (Kgms)	FLEXIBILITY GE IMPACT (% Elong.)	JP-5 FUEL (77 F)	MIL-H-83282 (150 F)	MIL-L-23699 (250 F)	DI-WATER (77 F)
TT-P-28-#1	2.5	F	P / F	1.0	1	P	P	P	F
TT-P-28-#2	1.25	P	P / P	<.5	1	P	P	P	P
A/Z-8-2/2	1.0	P	P / P	2.0	60	P	P	P	P
A/Z-22-2/2	1.0	P	P / P	1.0	40	P	P	P	P
A/Z-25-2/2	1.0	P	P / P	1.0	60	P	P	P	P
HTC-1	2.5	P	P / P	<.5	1/2	P	P	P	P
HTC-2	1.5	P	P / P	0.5	20	P	P	P	P

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TABLE XVI CORROSION RESISTANCE TEST RESULTS (5 DAY - 700F Cycle)

TEST CONDITIONS:

ASTM #B 117, 5% NaCl SALT SPRAY

SUBSTRATE: UNPRETREATED 1020 STEEL

500 HOURS EXPOSURE

NADC COATING IDENTIFICATION	SCRIBE AREA	GENERAL COATING SURFACE
TT-P-28-#1	5 - WHR	Many spots of corrosion over the surfaces of both panels with excessive rundown
TT-P-28-#2	5 - WHR	Many spots of corrosion over the surfaces of both panels with excessive rundown
A/Z-8-2/2	2 - WR	A few spots of corrosion on both panel surfaces with slight rundown
A/Z-22-2/2	2	A few small spots of corrosion on both panel surfaces with no rundown
A/Z-25-2/2	1 , 2	Virtually no evidence of corrosion on the surface of either panel
HTC-#1	5 - WHR	Surfaces of both panels were completely corroded as bad as the blank
HTC-#2	5 - WHR	Surfaces of both panels were completely corroded as bad as the blank

SCRIBE RATINGS:

1 - No corrosion in scribe

2 - Slight corrosion in scribe

3 - Some corrosion in scribe

4 - Moderate corrosion in scribe

5 - Heavy corrosion in scribe

WR - With rundown from scribe

WHR - With heavy rundown from scribe

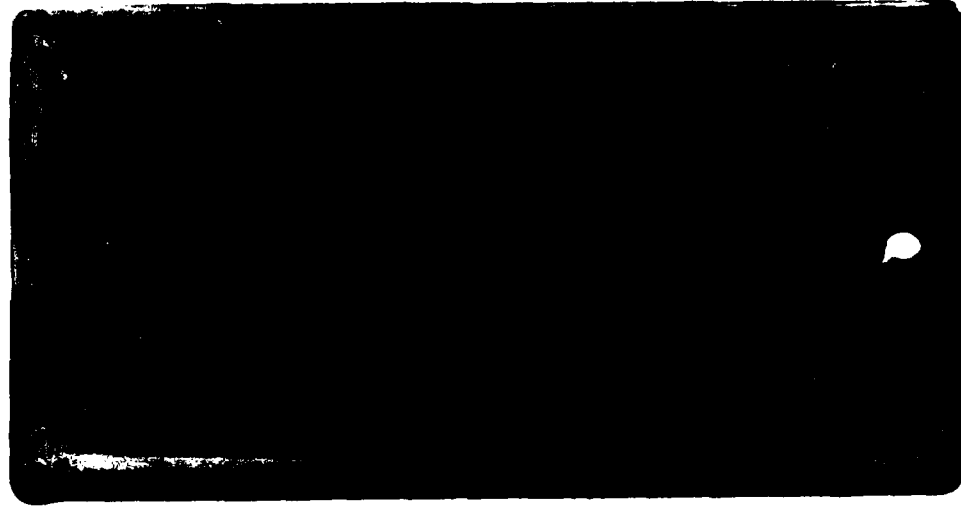
EXPOSURE: 500 HRS IN 5% NaCl SALT SPRAY

**PRE-CONDITIONING: 120 HRS THERMAL CYCLING BETWEEN ROOM
TEMPERATURE AND 700°F**

SUBSTRATE: BARE 1020 STEEL



**NADC
DEVELOPMENTAL
COATING**



TT-P-28



UNCOATED

FIGURE 4. TEST PANELS FOR CORROSION RESISTANCE TEST.

TABLE XVII COMPARISON OF HIGH TEMPERATURE COATINGS (1 HOUR @ 700 F)

COATING IDENTIFICATION	DRY-HARD TIME (Hrs)	THERMAL EXPOSURE 1 Hour @ 700F	ADHESION TESTS DRY/WET TAPE	ADHESION TESTS SCRAPE (Kgs)	FLEXIBILITY GE IMPACT (% Elong.)	FLUID RESISTANCE JP-5 FUEL (77 F) MIL-H-83282 (150 F) MIL-L-23699 (250 F) DI-WATER (77 F)
TT-P-28-#1	2.5	P	F / F	---	---	---
TT-P-28-#2	1.25	P	F / F	< 5	5	P P P P
A/Z-8-2/2	1.0	P	* / P	1.0	40	P P P P
A/Z-22-2/2	1.0	P	* / *	1.0	10	P P P P
A/Z-25-2/2	1.0	P	* / P	1.0	20	P P P P
HTC-1	2.5	P	P / P	0.5	< 0.5	P P P P
HTC-2	1.5	P	P / P	1.0	60	P P P P

* SLIGHT REMOVAL OF SOME OF THE COATING'S SURFACE

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TABLE XVIII CORROSION RESISTANCE TEST RESULTS (1 HOUR @ 700 F)

TEST CONDITIONS:

ASTM #B 117, 5% NaCl SALT SPRAY

SUBSTRATE: UNPRETREATED 1020 STEEL

500 HOURS EXPOSURE

NADC COATING IDENTIFICATION	SCRIBE AREA	GENERAL COATING SURFACE
TT-P-28-#1	FAILED THERMAL CYCLING - NOT TESTED	
TT-P-28-#2	5 - WHR	Many spots of corrosion over the surfaces of both panels with very fine rundown
A/Z-8-2/2	4 - WR	Many small spots of corrosion on both panel surfaces with some light rundown
A/Z-22-2/2	4 - WR	Some small spots of corrosion on the surfaces of both panels with some light rundown
A/Z-25-2/2	3 - WR (Slight)	Some small spots of corrosion on the surfaces of both panels with some light rundown
HTC-#1	5 - WHR	Surfaces of both panels were completely corroded as bad as the blank
HTC-#2	5 - WHR	Surfaces of both panels were completely corroded as bad as the blank

SCRIBE RATINGS:

- | | |
|-------------------------------|--------------------------------------|
| 1 - No corrosion in scribe | 2 - Slight corrosion in scribe |
| 3 - Some corrosion in scribe | 4 - Moderate corrosion in scribe |
| 5 - Heavy corrosion in scribe | |
| WR - With rundown from scribe | WHR - With heavy rundown from scribe |

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In the five day 400/500°F thermal series, the physical test results were approximately the same as those obtained for the five day 700°F thermal series, with the exception that both TT-P-28 samples passed the thermal cycling test. Again, the developed coatings performed the best in these tests, as can be seen in Table XIX. Also, the results obtained for the corrosion resistance test, shown in Table XX, were comparable to the ones for the one hour at 700°F series.

Finally, the coatings were evaluated after one week of room temperature exposure and no thermal conditioning. This data shown in Table XXI, indicated that although the test results varied slightly, there were no real significant differences between the physical properties of the materials. However, one difference between the results for this condition and the other thermal conditions was that all of the coatings softened to some degree in the fluid resistance test. Information for the corrosion test appears in Table XXII. The industry coatings, again, were as bad as the uncoated blank panels in these tests. The scribe areas for the TT-P-28 were similar to the HTC's, while the general surface areas were slightly better than the HTC's in appearance. The scribe areas for the in-house coatings were better than the other films, although, the general surface areas were only comparable to the TT-P-28 materials.

In general, throughout all of the tests under all of the conditions in this investigation, the NADC coating outperformed both the TT-P-28 coatings and the industry high temperature coatings. In this investigation, some interesting trends were apparent from analyzing all of the test data. The most significant trend is that the more severe the thermal conditioning became, the better the developed coating performed. Before thermal exposure, its performance is comparable with the TT-P-28 coatings, but with time and exposure, the experimental coatings performance far exceeded that of the other coatings. For the TT-P-28, however, only some of the properties improved with thermal conditioning, while the corrosion resistance decreased with severity of exposure. Finally, the industry high temperature materials steadily decreased in performance with exposure.

CONCLUSIONS

The developed coating has demonstrated improved performance in laboratory tests over the currently-used materials. It can be spray-applied to metallic substrates and provides excellent corrosion protection. In addition, the ease of application and ability to be used without a high temperature cure makes this coating a practical alternative to the standard TT-P-28 and Germatel W high-temperature materials.

FUTURE EFFORTS

Service testing of this material is planned to begin in the last quarter of 1987 and continue through to 1989. Upon the successful completion of the field evaluation, the developed coating will be transitioned into fleet use through the preparation of a military specification.

TABLE XIX COMPARISON OF HIGH TEMPERATURE COATINGS (5 DAY - 400/500F CYCLE)

COATING IDENTIFICATION	DRY HARD TIME (Hrs)	THERMAL CYCLING 5 DAY 70-400/500F	ADHESION TESTS DRY/WET TAPE	FLEXIBILITY GE IMPACT (% Elong.)	FLUID RESISTANCE			
					JP-5 FUEL (77 F)	MIL-H-83282 (150 F)	MIL-L-23699 (250 F)	DI-WATER (77 F)
TT-P-28-#1	2.5	P	* / P <.5	1	P	P	P	P
TT-P-28-#2	1.25	P	P / P <.5	1	P	P	P	P
A/Z-8-2/2	1.0	P	P / P 1.0	60	P	P	P	P
A/Z-22-2/2	1.0	P	P / P 1.0	60	P	P	P	P
A/Z-25-2/2	1.0	P	P / P 1.0	60	P	P	P	P
HTC-1	2.5	P	P / P 0.5	<0.5	F	P	P	P
HTC-2	1.5	P	P / P 0.5	20	P	P	P	P

* SLIGHT REMOVAL OF SOME OF THE COATING'S SURFACE

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TABLE XX CORROSION RESISTANCE TEST RESULTS (5 DAY - 400/500 F)

TEST CONDITIONS:

ASTM #B 117, 5% NaCl SALT SPRAY

SUBSTRATE: UNPRETREATED 1020 STEEL

500 HOURS EXPOSURE

NADC COATING IDENTIFICATION	SCRIBE AREA	GENERAL COATING SURFACE
TT-P-28-#1	5 - WHR	Many spots of corrosion over the surfaces of both panels with very fine rundown
TT-P-28-#2	5 - WHR	Many spots of corrosion over the surfaces of both panels with very fine rundown
A/Z-8-2/2	4 - WR	Many small spots of corrosion on both panel surfaces with some light rundown
A/Z-22-2/2	4 - WR	Small spots of corrosion on the surfaces of both panels with some light rundown
A/Z-25-2/2	3,4 - WR	Small spots of corrosion on the surfaces of both panels with some light rundown
HTC-#1	5 - WHR	Surfaces of both panels were completely corroded as bad as the blank
HTC-#2	5 - WHR	Surfaces of both panels were completely corroded as bad as the blank

SCRIBE RATINGS:

1 - No corrosion in scribe

2 - Slight corrosion in scribe

3 - Some corrosion in scribe

4 - Moderate corrosion in scribe

5 - Heavy corrosion in scribe

WR - With light rundown from scribe

WHR - With heavy rundown from scribe

TABLE XXI COMPARISON OF HIGH TEMPERATURE COATINGS (7 DAYS @ 70 F)

COATING IDENTIFICATION	DRY-HARD TIME (Hrs)	NO THERMAL CYCLING (77 F)	ADHESION TESTS DRY/WET TAPE	FLEXIBILITY GE IMPACT (% Elong.)	FLUID RESISTANCE			
					JP-5 FUEL (77 F)	MIL-H-83282 (150 F)	MIL-L-23699 (250 F)	DI-WATER (77 F)
TT-P-28-#1	2.5	---	P / P 2.0	20	*	*	*	*
TT-P-28-#2	1.25	---	P / P 2.0	20	*	*	*	*
A/Z-8-2/2	1.0	---	P / P 1.0	60	*	*	*	*
A/Z-22-2/2	1.0	---	P / P 0.5	60	*	*	*	*
A/Z-25-2/2	1.0	---	P / P 0.5	60	*	*	*	*
HTC-1	2.5	---	P / P <0.5	10	*	*	*	*
HTC-2	1.5	---	P / F 2.0	20	*	*	*	*

* ALL COATINGS SOFTENED TO SOME DEGREE

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TABLE XXII CORROSION RESISTANCE TEST RESULTS (7 DAYS @ 70 F)

TEST CONDITIONS:

ASTM #B 117, 5% NaCl SALT SPRAY

SUBSTRATE: UNPRETREATED 1020 STEEL

500 HOURS EXPOSURE

NADC COATING IDENTIFICATION	SCRIBE AREA	GENERAL COATING SURFACE
TT-P-28-#1	5 - WHR	Many spots of corrosion covering the entire surfaces of both panels with very fine rundown
TT-P-28-#2	5 - WHR	Many spots of corrosion covering the entire surfaces of both panels with very fine rundown
A/Z-3-2/2	5 - WR	Many small spots of corrosion on both panel surfaces with some light rundown
A/Z-22-2/2	5 - WR	Many small spots of corrosion on the surfaces of both panels with some light rundown
A/Z-25-2/2	4 - WR	Many small spots of corrosion on the surfaces of both panels with some light rundown
HTC-#1	5 - WHR	Surfaces of both panels were completely corroded as bad as the blank
HTC-#2	5 - WHR	Surfaces of both panels were completely corroded as bad as the blank

SCRIBE RATINGS:

1 - No corrosion in scribe

2 - Slight corrosion in scribe

3 - Some corrosion in scribe

4 - Moderate corrosion in scribe

5 - Heavy corrosion in scribe

WR - With rundown from scribe

WHR - With heavy rundown from scribe

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